The Absolute Stereochemistry of Ibhayinol from a South African Sea Hare

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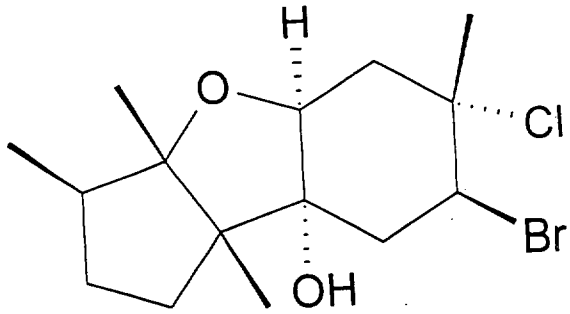
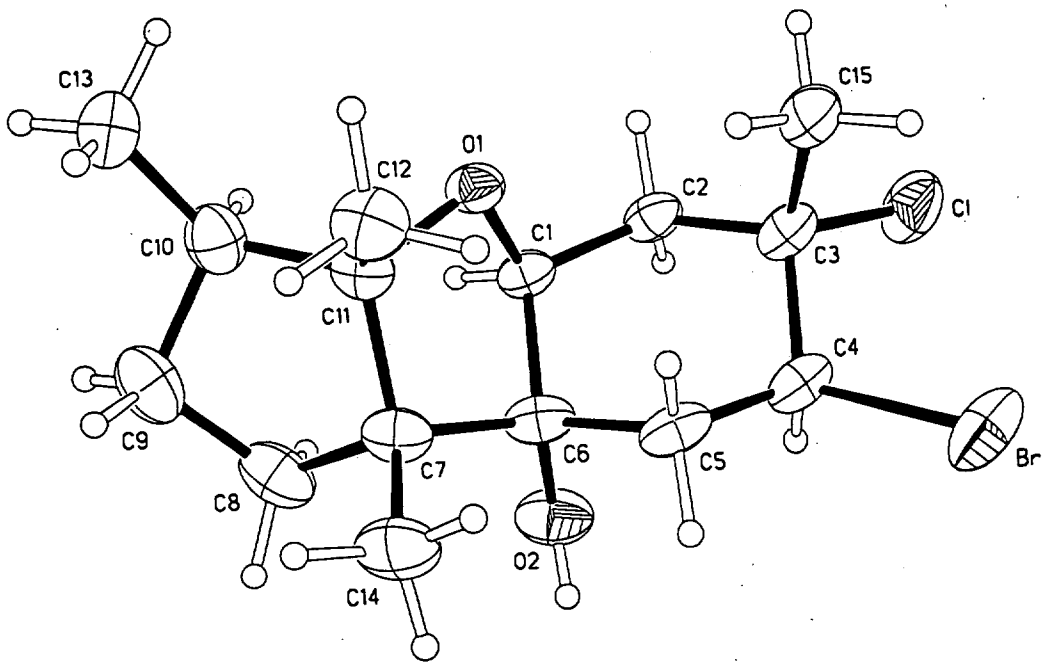
Supplementary material relating to the crystal structure determination of ibhayinol [5].





Anisotropic atomic displacement ellipsoids for the non-hydrogen atoms are shown at the 50% probability level. Only one component of the hydroxyl group disorder is shown. Hydrogen atoms are displayed with an arbitrarily small radius.

Table 1. Sample and crystal data for 5.

Crystallization solvents	Ethyl acetate and hexane	
Crystallization method	Slow evaporation	
Crystal habit	Colourless plate	
Crystal size	0.13 x 0.12 x 0.03 mm	
Empirical formula	$C_{15}H_{24}BrClO_2$	
Formula weight	351.70	
Temperature	150(2) K	
Wavelength	1.54178 Å	
Crystal system	Orthorhombic	
Space group	I222	
Unit cell dimensions	a = 12.4352(14) Å	α= 90°
	b = 12.7135(14) Å	β= 90°
	c = 20.648(2) Å	γ = 90°
Volume	3264.3(11) $Å^3$	
Z	8	
Density (calculated)	1.431 Mg/m^3	
Absorption coefficient	4.905 mm^{-1}	
F(000)	1456	

Table 2. Data collection and structure refinement for 5.

Diffractometer	Bruker AXS SMART 6000
Radiation source	Normal focus sealed tube, CuK_α
Data collection method	ω scans
Theta range for data collection	4.08 to 73.03°
Index ranges	$-15 \leq h \leq 13, -12 \leq k \leq 15, -22 \leq l \leq 24$
Reflections collected	12419
Independent reflections	3207 [R(int) = 0.0503]
Coverage of independent reflections	98.9 %
Variation in check reflections	N/A
Absorption correction	Integration
Max. and min. transmission	0.8597 and 0.5834
Structure solution technique	Direct methods
Structure solution program	SHELXTL V5.10 UNIX (Bruker, 1997)
Refinement technique	Full-matrix least-squares on F^2
Refinement program	SHELXTL V5.10 UNIX (Bruker, 1997)
Function minimized	$\Sigma w(F_o^2 - F_c^2)^2$
Data / restraints / parameters	3207 / 2 / 183
Goodness-of-fit on F^2	0.997
Δ/σ_{max}	1.195
Final R indices	
2948 data; $I>2\sigma(I)$	R1 = 0.0296, wR2 = 0.0664
all data	R1 = 0.0327, wR2 = 0.0674
Weighting scheme	$w = 1/[\sigma^2(F_o^2) + (0.0346P)^2]$ where $P = [MAX(F_o^2, 0) + 2F_c^2]/3$
Absolute structure parameter	0.000(17)
Largest diff. peak and hole	0.446 and -0.395 eÅ^{-3}

Table 3. Atomic coordinates and equivalent isotropic atomic displacement parameters (Å^2) for 5.

U(eq) is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x/a	y/b	z/c	U(eq)
C1	0.19698(19)	0.34645(19)	0.47899(12)	0.0264(5)
C2	0.15829(19)	0.3226(2)	0.54730(12)	0.0309(5)
C3	0.1909(2)	0.2147(2)	0.57408(13)	0.0349(6)
C4	0.3112(2)	0.1999(2)	0.56167(13)	0.0337(6)
C5	0.3382(2)	0.2081(2)	0.49004(14)	0.0340(6)
C6	0.3143(2)	0.3182(2)	0.46420(13)	0.0308(5)
C7	0.3167(2)	0.3254(2)	0.38917(13)	0.0321(5)
C8	0.3311(2)	0.4400(3)	0.36495(14)	0.0438(6)
C9	0.2550(3)	0.4492(3)	0.30768(15)	0.0455(7)
C10	0.1548(3)	0.3917(2)	0.33054(13)	0.0393(6)
C11	0.1974(2)	0.2963(2)	0.37022(12)	0.0304(5)
C12	0.1822(3)	0.1913(2)	0.33702(14)	0.0404(6)
C13	0.0766(3)	0.3671(3)	0.27605(16)	0.0545(9)
C14	0.4024(3)	0.2574(3)	0.35697(16)	0.0450(7)
C15	0.1197(3)	0.1263(2)	0.54993(15)	0.0425(7)
O1	0.13908(15)	0.28827(14)	0.43173(8)	0.0285(3)
O2	0.38600(18)	0.38934(18)	0.49632(11)	0.0432(5)
Cl	0.17206(7)	0.22285(7)	0.66135(4)	0.0507(2)
Br	0.36594(3)	0.06540(2)	0.594315(16)	0.04860(10)

Table 4. Selected bond lengths (Å) for 5.

C1-O1	1.421(3)	C1-C2	1.521(4)
C1-C6	1.533(3)	C2-C3	1.534(4)
C3-C15	1.515(4)	C3-C4	1.529(4)
C3-Cl	1.820(3)	C4-C5	1.520(4)
C4-Br	1.960(3)	C5-C6	1.528(4)
C6-O2	1.432(3)	C6-C7	1.552(4)
C7-C14	1.525(4)	C7-C8	1.552(4)
C7-C11	1.578(4)	C8-C9	1.519(5)
C9-C10	1.521(4)	C10-C13	1.520(4)
C10-C11	1.557(4)	C11-O1	1.466(3)
C11-C12	1.513(3)		

Table 5. Selected bond angles (°) for 5.

O1-C1-C2	111.9(2)	O1-C1-C6	102.92(18)
C2-C1-C6	116.1(2)	C1-C2-C3	115.4(2)
C15-C3-C4	115.1(2)	C15-C3-C2	113.0(2)
C4-C3-C2	108.0(2)	C15-C3-Cl	107.0(2)
C4-C3-Cl	107.39(18)	C2-C3-Cl	105.78(18)
C5-C4-C3	111.8(2)	C5-C4-Br	108.51(18)
C3-C4-Br	112.9(2)	C4-C5-C6	111.1(2)
O2-C6-C5	107.2(2)	O2-C6-C1	110.6(2)
C5-C6-C1	109.3(2)	O2-C6-C7	114.4(2)
C5-C6-C7	113.5(2)	C1-C6-C7	101.7(2)
C14-C7-C8	108.2(2)	C14-C7-C6	114.5(2)
C8-C7-C6	112.3(2)	C14-C7-C11	114.6(2)
C8-C7-C11	104.4(2)	C6-C7-C11	102.5(2)
C9-C8-C7	104.6(2)	C8-C9-C10	103.4(2)
C13-C10-C9	113.2(3)	C13-C10-C11	116.6(3)
C9-C10-C11	105.0(3)	O1-C11-C12	105.6(2)
O1-C11-C10	110.0(2)	C12-C11-C10	114.0(2)
O1-C11-C7	105.48(19)	C12-C11-C7	115.9(2)
C10-C11-C7	105.6(2)	C1-O1-C11	107.92(18)

Table 6. Selected torsion angles (°) for 5.

O1-C1-C2-C3	73.1(3)	C6-C1-C2-C3	-44.6(3)
C1-C2-C3-C15	-80.3(3)	C1-C2-C3-C4	48.2(3)
C1-C2-C3-Cl	162.93(18)	C15-C3-C4-C5	69.3(3)
C2-C3-C4-C5	-58.0(3)	Cl-C3-C4-C5	-171.66(19)
C15-C3-C4-Br	-53.4(3)	C2-C3-C4-Br	179.28(16)
Cl-C3-C4-Br	65.6(2)	C3-C4-C5-C6	64.1(3)
Br-C4-C5-C6	-170.68(17)	C4-C5-C6-O2	65.1(3)
C4-C5-C6-C1	-54.9(3)	C4-C5-C6-C7	-167.6(2)
O1-C1-C6-O2	165.6(2)	C2-C1-C6-O2	-71.9(3)
O1-C1-C6-C5	-76.6(2)	C2-C1-C6-C5	45.9(3)
O1-C1-C6-C7	43.6(2)	C2-C1-C6-C7	166.2(2)
O2-C6-C7-C14	86.8(3)	C5-C6-C7-C14	-36.7(3)
C1-C6-C7-C14	-154.0(2)	O2-C6-C7-C8	-37.1(3)
C5-C6-C7-C8	-160.6(2)	C1-C6-C7-C8	82.2(3)
O2-C6-C7-C11	-148.5(2)	C5-C6-C7-C11	88.0(2)
C1-C6-C7-C11	-29.2(2)	C14-C7-C8-C9	93.8(3)
C6-C7-C8-C9	-138.8(2)	C11-C7-C8-C9	-28.6(3)
C7-C8-C9-C10	41.5(3)	C8-C9-C10-C13	-166.1(3)
C8-C9-C10-C11	-37.8(3)	C13-C10-C11-O1	-100.8(3)
C9-C10-C11-O1	133.1(2)	C13-C10-C11-C12	17.5(4)
C9-C10-C11-C12	-108.6(3)	C13-C10-C11-C7	145.9(3)
C9-C10-C11-C7	19.7(3)	C14-C7-C11-O1	130.8(2)
C8-C7-C11-O1	-111.1(2)	C6-C7-C11-O1	6.1(3)
C14-C7-C11-C12	14.5(4)	C8-C7-C11-C12	132.6(2)
C6-C7-C11-C12	-110.2(2)	C14-C7-C11-C10	-112.7(3)
C8-C7-C11-C10	5.4(3)	C6-C7-C11-C10	122.6(2)
C2-C1-O1-C11	-166.7(2)	C6-C1-O1-C11	-41.4(2)
C12-C11-O1-C1	145.1(2)	C10-C11-O1-C1	-91.5(2)
C7-C11-O1-C1	21.9(2)		

Table 7. Anisotropic atomic displacement parameters ($Å^2$) for 5.
The anisotropic atomic displacement factor exponent takes the form:
$-2\pi^2 [h^2a^{*2}U_{11} + ... + 2hka^* b^* U_{12}]$

	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
C1	0.0223(12)	0.0286(11)	0.0283(12)	-0.0068(9)	-0.0019(9)	0.0022(9)
C2	0.0228(12)	0.0398(13)	0.0302(12)	-0.0060(9)	-0.0015(9)	0.0073(10)
C3	0.0305(13)	0.0417(14)	0.0325(13)	-0.0027(10)	-0.0093(10)	0.0054(12)
C4	0.0292(13)	0.0311(12)	0.0409(14)	-0.0081(10)	-0.0127(11)	0.0064(11)
C5	0.0226(13)	0.0340(12)	0.0453(14)	-0.0115(10)	-0.0067(10)	0.0071(10)
C6	0.0219(12)	0.0326(13)	0.0378(14)	-0.0138(10)	-0.0008(10)	0.0003(10)
C7	0.0264(12)	0.0321(12)	0.0377(14)	-0.0116(10)	0.0044(10)	-0.0006(10)
C8	0.0453(16)	0.0394(14)	0.0466(15)	-0.0091(13)	0.0128(12)	-0.0126(14)
C9	0.0588(19)	0.0374(16)	0.0402(15)	-0.0028(11)	0.0134(13)	-0.0042(15)
C10	0.0451(18)	0.0394(13)	0.0335(14)	0.0005(10)	0.0060(11)	0.0028(13)
C11	0.0300(13)	0.0318(12)	0.0295(12)	-0.0076(9)	0.0034(9)	-0.0002(11)
C12	0.0447(16)	0.0383(14)	0.0381(14)	-0.0128(11)	-0.0022(12)	-0.0058(13)
C13	0.0506(19)	0.073(2)	0.0399(17)	0.0173(15)	-0.0058(14)	-0.0015(18)
C14	0.0322(14)	0.0547(18)	0.0482(16)	-0.0138(13)	0.0089(12)	0.0049(13)
C15	0.0377(16)	0.0467(16)	0.0430(15)	0.0063(11)	-0.0128(12)	-0.0040(14)
O1	0.0235(8)	0.0360(8)	0.0260(8)	-0.0042(6)	-0.0012(7)	-0.0018(8)
O2	0.0328(11)	0.0479(10)	0.0487(12)	-0.0165(9)	-0.0040(8)	-0.0121(10)
Cl	0.0516(5)	0.0698(5)	0.0306(3)	0.0029(3)	-0.0066(3)	0.0133(4)
Br	0.05234(18)	0.03658(13)	0.05687(18)	-0.00750(14)	-0.02546(15)	0.01470(14)

Table 8. Hydrogen atom coordinates and isotropic atomic displacement parameters ($Å^2$) for 5.

	x/a	y/b	z/c	U
H1A	0.1864	0.4231	0.4703	0.032
H2A	0.0788	0.3276	0.5480	0.037
H2B	0.1865	0.3776	0.5767	0.037
H4A	0.3503	0.2575	0.5848	0.040
H5A	0.2955	0.1558	0.4655	0.041
H5B	0.4153	0.1918	0.4835	0.041
H8A	0.3117	0.4910	0.3993	0.053
H8B	0.4063	0.4529	0.3514	0.053
H9A	0.2858	0.4154	0.2687	0.055
H9B	0.2390	0.5238	0.2978	0.055
H10A	0.1167	0.4393	0.3615	0.047
H12A	0.2114	0.1353	0.3645	0.061
H12B	0.2200	0.1917	0.2954	0.061
H12C	0.1053	0.1789	0.3297	0.061
H13A	0.0566	0.4324	0.2539	0.082
H13B	0.0120	0.3339	0.2940	0.082
H13C	0.1107	0.3191	0.2451	0.082
H14A	0.3994	0.2672	0.3099	0.068
H14B	0.3893	0.1833	0.3674	0.068
H14C	0.4736	0.2780	0.3729	0.068
H15A	0.0444	0.1429	0.5594	0.064
H15B	0.1396	0.0606	0.5717	0.064
H15C	0.1292	0.1181	0.5031	0.064
H2 *	0.450(2)	0.372(6)	0.491(5)	0.065
H2' *	0.376(7)	0.4538(16)	0.491(4)	0.065

* Site occupancy factor is 0.5 for these hydrogen atoms.

Table 9. Selected hydrogen bond information for 5 (Å and °).

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
O2-H2...O2#1	0.838(10)	2.06(4)	2.839(5)	154(8)
O2-H2'...O2#2	0.836(10)	2.02(3)	2.818(5)	161(9)

#1 -x+1,y,-z+1 #2 x,-y+1,-z+1